ANL-ART-153

Fiscal Year 2018 Sodium-CO_2 Interaction Test

Nuclear Science and Engineering Division

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Fiscal Year 2018 Sodium-CO_2 Interaction Test

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ABSTRACT

An experiment campaign was continued in Fiscal Year 2018 in the Argonne National Laboratory (Argonne) SNAKE facility that investigated the interactions between supercritical carbon dioxide (sCO_2) and sodium through injection of sCO_2 into an open surface sodium pool. Two of fifteen previous SNAKE tests revealed that self-plugging of the leakage path/nozzle occurs at higher initial sodium temperatures of about 500 ºC due to chemical reactions involving CO_2, sodium, and sodium-CO_2 reaction products. At lower sodium temperatures, the interaction phenomena involve consumption of injected CO_2 and formation of an agglomerated mass of reaction products without self-plugging. The FY 2018 test investigated interactions at an intermediate initial sodium temperature of about 411 ºC, representative of the interior of a sodium-to-CO_2 heat exchanger in a Sodium-Cooled Fast Reactor (SFR) sCO_2 Brayton Cycle energy conversion system. The experiment was successfully conducted and did not show self-plugging as evidenced by a sustained chemical reaction between the injected CO_2 and sodium and consumption of CO_2.

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Table of Contents

EXECUTIVE SUMMARY

The following provides a summary of the work performed at the Argonne National Laboratory (ANL) sodium – supercritical carbon dioxide (sCO_2) interaction facility, SNAKE, during Fiscal Year 2018.

Development of the sCO_2 Brayton cycle as an advanced power converter for use with Sodium-Cooled Fast Reactors (SFRs) as well as other Advanced Reactors has been underway at Argonne National Laboratory since 2002 under the Nuclear Technology Research and Development (NTRD) and predecessor U.S. DOE Programs and Initiatives. The main benefits of sCO_2 power conversion for SFRs are improved economics (reduction of the Nuclear Power Plant overnight capital cost per unit output electrical power, \$/kWe, or, alternately, the Levelized Cost of Electricity, LCOE) and the elimination of the need to accommodate potential sodium-water reactions. However, sodium and carbon dioxide (CO_2) interact chemically such that there is a need to understand and accommodate potential sodium-CO_2 interactions.

The expectation is that sodium-CO_2 interactions under prototypical reactor conditions will be relatively benign compared with sodium-water reactions. If sodium and sCO_2 come into contact and interact, it will be inside of a sodium-to-CO_2 heat exchanger. Due to the heat transfer properties of CO_2, there is an incentive to utilize compact diffusion-bonded sodium-to-sCO_2 heat exchangers to reduce the heat exchanger size and cost relative to traditional shell-and-tube technology. Thus, compact diffusion-bonded sodium-to-sCO_2 heat exchanger designs define the prototypical conditions of interest to the study of sodium-sCO_2 interactions. Two postulated failure locations are inside the monolithic structure of the diffusion-bonded core, and at the headers welded to the diffusion-bonded core. In the event of a postulated crack inside the core, sCO_2 could be released into a single sodium channel. The CO_2 could displace sodium from the channel and discharge into the larger open plenum volumes of the sodium headers welded to the diffusion-bonded core. In the event of a postulated crack at the header weld, CO_2 would be released directly into the open header plenum volume. Data on sodium-sCO_2 interactions under prototypical conditions in such configurations did not exist prior to SNAKE experiments. The Argonne sodium-sCO_2 interaction experiments in the SNAKE facility have been designed to simulate prototypical interaction conditions in these two configurations.

Because a postulated structural failure of a compact diffusion-bonded heat exchanger core is expected to correspond to a micro-crack, the sCO_2 release rate into sodium is expected to be limited which corresponds to a low exothermic energy release rate. However, sodium-CO_2 interactions can result in the formation of solid reaction products such as sodium carbonate that do not readily undergo dissolution in sodium. Reaction products could accumulate in the intermediate sodium heat transport system, significantly affecting performance and requiring cleanup. A highly interesting phenomenon is that a micro-crack could, under the right circumstances, plug itself due to the reactions of sodium, CO_2, and sodium-CO_2 reaction products at the micro-crack itself. This plug would seal the crack opening and stop the release of CO_2. If this occurs, the extent of sodium-CO_2 interaction would be limited to only a minor amount by this self-plugging phenomenon. These are some of the phenomena under investigation in the Argonne sodium-sCO_2 interaction experiments in the SNAKE facility.

The top-level goal of SNAKE is to gain a fundamental understanding of sodium-sCO_2 interactions, with a focus on the specific scenarios of the failure of a compact diffusion-bonded sodium-CO_2 heat exchanger (DBHE).

The SNAKE experiment program was initiated in Fiscal Year 2010 and began with a study of prototypic conditions during failure, identification of primary scaling parameters, and the design basis for the test facility. Several previous reports described the facility scaling rationale and design. Assembly of SNAKE began in Fiscal Year 2011 and was completed in July 2012. The maximum sodium inventory in this facility is 33 lbs (15 kg; ~15.5 L/4.1 gal).

The first sodium-sCO_2 interaction shakedown experiment was carried out at SNAKE in September 2012 and a series of sodium-sCO_2 interaction experiments were carried out in Fiscal Year 2013. These tests successfully injected supercritical carbon dioxide into a pool of sodium through a 64 µm diameter nozzle. A reaction between the CO_2 and sodium was detected. The chemical reaction was unexpected since the initial sodium temperature was 145 ˚C, a temperature range in which previous studies detected little or no chemical reaction. The important difference between the SNAKE experiments and previous research was that the SNAKE geometry accurately reflects actual DBHE designs and was able to capture conditions that promote high-interfacial area and mixing between the CO_2 and sodium. These features were likely important in promoting accelerated chemical reactions and will continue to be studied as the SNAKE test matrix is carried out. Since the original test vessel and dump tank configuration had a complex geometry that made cleaning of reaction products challenging, simplified vessels were designed and procured in Fiscal Year 2014. The new SNAKE apparatus design philosophy was simplicity and interchangeability. Progressively higher temperature testing (330 °C) was carried out in Fiscal Year 2014 and confirmed that the sodium-sCO_2 interaction consumed CO_2, was exothermic and produced carbon monoxide. Even higher temperature testing, up to 500 °C, was performed in Fiscal Year 2016 to capture the range of prototypic temperatures which was expected to be a key parameter controlling the extent of the sodium-sCO_2 reaction. In Fiscal Year 2017, a single experiment was performed in which CO_2 was injected into a mockup of a single semicircular sodium channel.

In Fiscal Year 2018, a single experiment was performed. Exp2018-16 was completed on September 7th 2018. The sodium temperature was 411°C, the CO_2 temperature was approximately 400°C, and the initial CO_2 injection pressure was 20 MPa. The CO_2 was injected through a 74 µm nozzle into an open pool of sodium approximately 0.85 m deep. The injection lasted 20 minutes.

Data from Exp2018_16 is reported and initial interpretations are discussed in this report.

1 Introduction

The Department of Energy's Office of Nuclear Energy (DOE/NE) Nuclear Technology Research and Development (NTRD) program is focused on the development of clean, safe, and affordable nuclear power. This program contains a spectrum of experiment facilities to support R&D on advanced reactor concepts including the SNAKE (S-CO_2 Na Kinetics Experiment) apparatus at Argonne National Laboratory (Argonne).

The supercritical carbon dioxide (sCO_2) Brayton cycle, coupled with a Sodium-Cooled Fast Reactor (SFR), has been identified as a new and innovative energy conversion technology that could contribute to meeting NTRD objectives especially in terms of improved economics and safety. One appealing feature of the sCO_2 Brayton energy conversion system is the smaller footprint that the hardware requires relative to the traditional superheated steam cycle, which is in part due to the small size of the turbomachinery as well as the use of compact diffusion-bonded heat exchangers (DBHE) as the heat source heat exchanger (sodium-to-CO_2) as well as the recuperator and cooler modules.

DBHEs have a high degree of structural integrity; however, the potential for leaks to develop between the sodium and CO_2 coolant channels in the secondary heat exchanger cannot be ruled out. This type of failure would lead to discharge of high pressure CO_2 into the secondary coolant circuit. Due to the robustness of the DBHE design, catastrophic failure leading to CO_2 jet blowdown into the intermediate sodium loop is not deemed likely. Rather, small cracks (or micro-leaks) may develop in which CO_2 will bleed into the secondary system at a relatively low rate and chemically react with the sodium.

In recognition of the anticipated failure mode for a DBHE sodium-to-CO_2 heat exchanger, an Argonne experiment program, SNAKE, was initiated in Fiscal Year 2010 (see Farmer et al., 2010; Gerardi et al. 2011, 2012a, 2012b, 2013a&b, 2014, 2015a&b, 2016, and 2017a&b). The purpose of the SNAKE program was to investigate the reaction characteristics between sodium and CO_2 under micro-leak conditions. Specifically, the objective of the SNAKE facility was to determine the fundamental nature and extent of the chemical reactions that occur when high-pressure CO_2 was injected into liquid sodium from a micro-leak across a stainless steel pressure boundary as a function of the sodium pool temperature and inlet CO_2 flowrate. SNAKE was also designed to examine the potential for the micro-leak to seal itself as a result of blockage formation from the chemical reaction byproducts of the sodium-CO_2 reaction, or as a result of oxide layer buildup on the crack faces. An additional objective added in FY 2016 was to develop and test an acoustic technique that could be used to detect gas (CO_2 or other high pressure gas) leaks into sodium.

An initial test matrix for fundamental sodium (Na)-carbon dioxide (CO_2) interactions was formulated to provide the data needed to understand sodium-CO_2 interactions and their consequences for SFRs incorporating supercritical CO_2 Brayton cycle power converters with compact diffusion-bonded sodium-to-CO_2 heat exchangers (Gerardi et al., 2013). This report provides an update on the single experiment performed in Fiscal Year 2018.

2 Experimental

The SNAKE test program follows the Argonne Quality Level C (QL-C) procedure (LMS-PROC-125) for programmatic, training, documents and records, procurement, and design. Each test is carried out according to a formal procedure that identifies the test objectives, and step-by-step operator actions. At the conclusion of each test, it is classified according to the following metrics:

> **Accepted data** – test was performed fully within scope and defined procedures; all critical instruments operated within specifications and returned data.
>
> **Scoping data** – some aspects of the test fell outside of the intended scope or procedures and/or some instruments did not operate within specifications or return data. Data could still be available for specific uses.
>
> **Reject data** – test was unsuccessful and outside of scope. Equipment, or instruments did not work as intended and some or all data is suspect.

2.1 Current Test Matrix

The general test matrix for SNAKE described in Gerardi et al. (2013) included a near-term test matrix to guide shakedown experiments. The Fiscal Year 2015 through 2017 experiments yielded excellent data on the extent of the sodium – sCO_2 reaction for prototypical conditions for a SFR coupled with a sCO_2 Brayton energy conversion cycle. These experiments were used to update the near-term test matrix to obtain high-quality data for modeling purposes with the smallest number of experiments.

A summary of the completed experiments and the target conditions of the next several experiments are listed in Table 1. All of these tests would be direct injection of CO_2 into a static sodium pool. Several tests were performed and may continue to be performed using inert N_2 to study non-reactive jets for comparison with the reactive jet test data. Low pressure (sub-critical) CO_2 tests may also be carried out to add to the simulation verification database. Most tests would last approximately 5-30 minutes.

The first experiment was completed in September 2012 and summarized in Gerardi et al. (2012b). The next seven tests were carried out in Fiscal Year 2013-2015 and detailed in Gerardi et al. (2013b, 2014, 2015b). Two series of inert gas jet tests in sodium were performed in Fiscal Year 2016 along with one sCO_2-sodium experiment (Gerardi et al., 2016).

A sCO_2-sodium test using a DBHE mock-up channel, Exp2016_15, was completed in FY 2017.

Note that the original SNAKE test matrix included experiments investigating the interaction of carbon monoxide with sodium. Those tests are no longer planned due to the extreme complexity of handling carbon monoxide. Some planning for these tests was completed in FY 2017 with the realization that substantial funding and resources would need to be dedicated to support the safety infrastructure and monitoring programs required to inject carbon monoxide into sodium.

Test #	Test Name	Test fiscal year	Data Quality	Test Gas	Gas initial stagnation pressure, MPa	Gas stagnation temperature, °C	Sodium column temperature, °C	Sodium column height, cm	Injected gas mass, g	Test duration, min	Nozzle Diameter (um)	Notes	Test completed
1	Exp2012_01	2013	Scoping	CO2 - Ultra High Purity	9	170	180	38.1	247	30	64	Some CO production; minimal exothermic	19-Sep-12
2	Exp2013_02	2013	Scoping	CO2 - Ultra High Purity	2.8	143	141-145	46.8	7.8	4.2	64	CO Production; exothermic; Solids	16-Aug-13
3	Exp2013_03	2013	Scoping	CO2 - Ultra High Purity	6.1	146	141-184	48.4	48.5	4.2	64	CO Production; exothermic; Solids	16-Aug-13
4	Exp2013_04	2013	Scoping	CO2 - Ultra High Purity	7.6	144	142-216	53.6	61.4	4.2	64	CO Production; exothermic; Solids	16-Aug-13
5	Exp2013_05	2013	Scoping	CO2 - Ultra High Purity	8.6	146	143-242	55.0	87.9	4.2	64	CO Production; exothermic; Solids	16-Aug-13
6	Exp2013_06	2013	Scoping	CO2 - Ultra High Purity	10.1	145	145-253	54.9	39.1	4.2	64	CO Production; exothermic; Solids	16-Aug-13
7	Exp2013_07	2013	Scoping	CO2 - Ultra High Purity	11.3	145	145-259	53.7	40.5	4.2	64	CO Production; exothermic; Solids	16-Aug-13
8	Exp2014_01	2014	Accept	CO2 - Research Grade	16	300	332	51.5	120	22	73	CO Production; exothermic; Solids; No CO2	18-Sep-14
9	Exp2015_01	2015	Accept	CO2 - Research Grade	16.9	356.8	369	27.5	~0	23.3	74	Plugged	20-Mar-15
10	Exp2015_02	2015	Reject (Na load)	CO2 - Research Grade	-	-	-	-	-	-	62	No sodium loaded	7-Jul-15
11	Exp2015_03	2015	Accept	CO2 - Research Grade	20.2	362-374	371-391	52.8	108	50	62	CO2 consumption; CO production; exothermic	29-Jul-15
12	N2_300um_SA	2016	Accept	N2	Range of temps and pressures for non-reactive gas jet studies			20.0-70.0	>10,000	20-73	280	Non-reactive jet; Acoustic detection	21-Dec-15
13	N2_75um_SB	2016	Accept	N2	Range of temps and pressures for non-reactive gas jet studies			40.0	>10,000	40	71	Non-reactive jet; Acoustic detection	13-Apr-16
14	Exp2016_14	2016	Accept	CO2 - Research Grade	20	495	499	40.0	<1	63	71	Plugged; Acoustic detection	14-Apr-16
15	Exp2016_15	2017	Accept	CO2 with DBHX Channel Geometry	16.1	328.3	332	59.3	455	34	115	High temp recorded; DBHX geometry; CO2 consumption; CO production; exothermic	1-Mar-17
16	Exp2018_16	2018	Accept	CO2 - Research Grade	20	400	420	85.3	58	20	74	CO2 consumption; CO production; exothermic	7-Sep-18
17		2018/2019 - planned		CO2 - Research Grade	20	450	450	20.0	-	20			
18		2019/2020 - planned		CO2 - Research Grade	16	480	500	50.0	-	20			
19		2019/2020 - planned		CO2 - Research Grade	20	300	330	50.0	-	20			

Table 1. Updated SNAKE test matrix – first 15 experiments in green were completed in 2013-2017. Experiment 16 is the primary topic of this report

3 Exp2018_16 sCO_2 Sodium Test

The objective of Exp2018_16 was to study the interaction of a sCO_2 leak into an open pool of sodium at an intermediate sodium temperature of about 420 ºC to determine if self-plugging occurs at this temperature. Upon completion of the test and review of the data, the experiment objectives were met.

3.1 Exp2018_16 Test Summary

This test injected supercritical CO_2 into an open pool of sodium for 20 min. This experiment simulated conditions prototypical of a SFR coupled with a sCO_2 Brayton energy conversion cycle with a leak inside the interior of the heat exchanger away from the ends. Exp2018_16 was designated as a "accept data" level test. The initial sCO_2 injection pressure and temperature were 20 MPa and 383.4 °C, respectively. The initial sodium temperature and height above the nozzle were 410.9 °C and 85.3 cm, respectively. The sCO_2 was injected through a well-characterized nozzle, which had a diameter of 74.0 µm ± 2 µm. The total CO_2 mass injected was approximately 58.0 g. The bulk sodium temperature rose approximately 21.0±2 ˚C during the test indicating an exothermic reaction.

A significant amount of data was generated during this test including fiber optic distributed temperature data, flow meter, pressure, and thermocouple measurements, etc.

Highlights of Exp2018_16 include:

- Initial sodium level was 85.3 cm above the injection point for a total sodium loading of 5.98 kg.
- The injection nozzle was 74 micron in diameter.
- Initial sodium temperature was 410.9 °C. The initial CO_2 reservoir tank pressure was ~20 MPa.
- Both CO_2 and CO were detected at the exhaust outlet but significant gas consumption did occur with solid reaction product production.
- The sodium temperature went up to approximately 432.8 °C. Periodically, higher temperature values were recorded, above 500 °C.

This test was another example of sCO_2 injection into sodium that did not plug the injection nozzle and an exothermic reaction was sustained that produced solid reaction products and some CO.

3.2 New Fiber Installation and Position

A new method of installing and capturing the DTS fiber sensor was attempted in this test versus previous SNAKE experiments. Previous installations revolved around one sensor installed from the top of the vessel hanging loosely to the near bottom. This fiber was used during sodium fill and was assumed to be forced to the outside wall during injection. This would limit its ability to accurately record the experiment as the temperatures of the inner wall and heaters would become the dominant temperature reading. A second DTS fiber would then be installed through the bottom hub. Complicated geometry and multiple layers of insulation often prevented a reliable signal from being obtained and many fibers were broken. The new method involved two fibers being installed from the top of the test vessel and inserted through guide tubes welded to the side of the injection nozzle. This method should capture the fiber in the reaction stream without adding strain and protecting it from breakage, as shown in Figure 1.

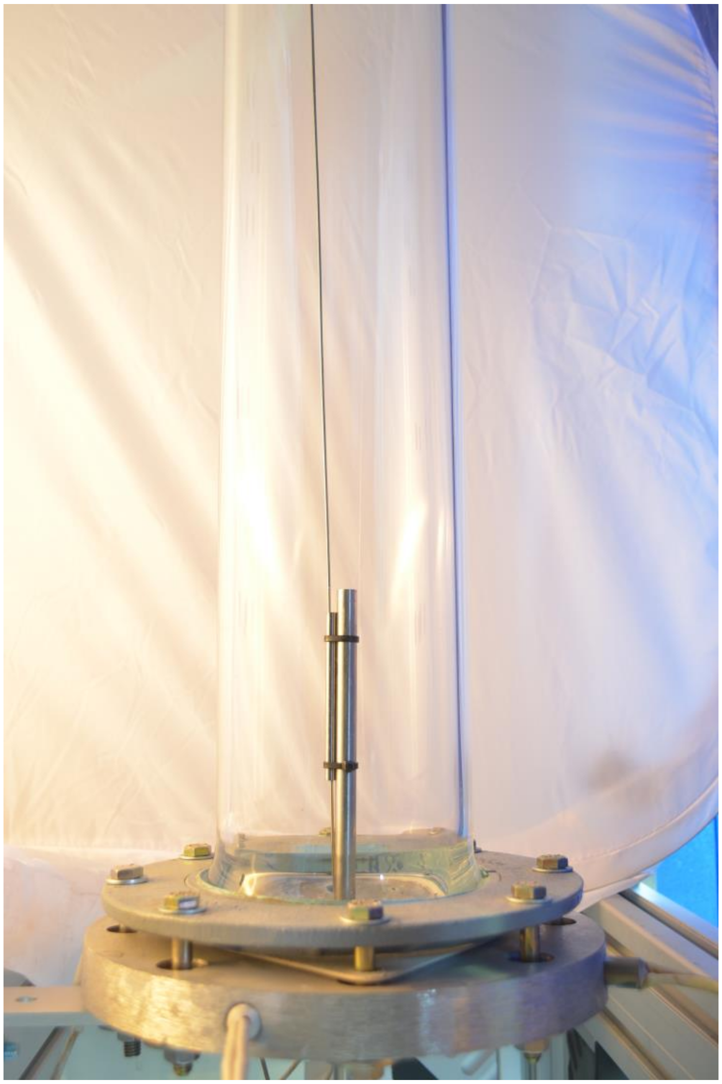


Figure 1. Mock-up showing new DTS fiber installation.

3.2.1 Design

The design of the injector assembly strove to maintain most of the design features and dimensions of previous SNAKE injectors. The injector needed to fit inside of a standard SNAKE test vessel and interface externally with existing sodium fill and drain lines, and the sCO_2 injection line, as shown in Figure 2.

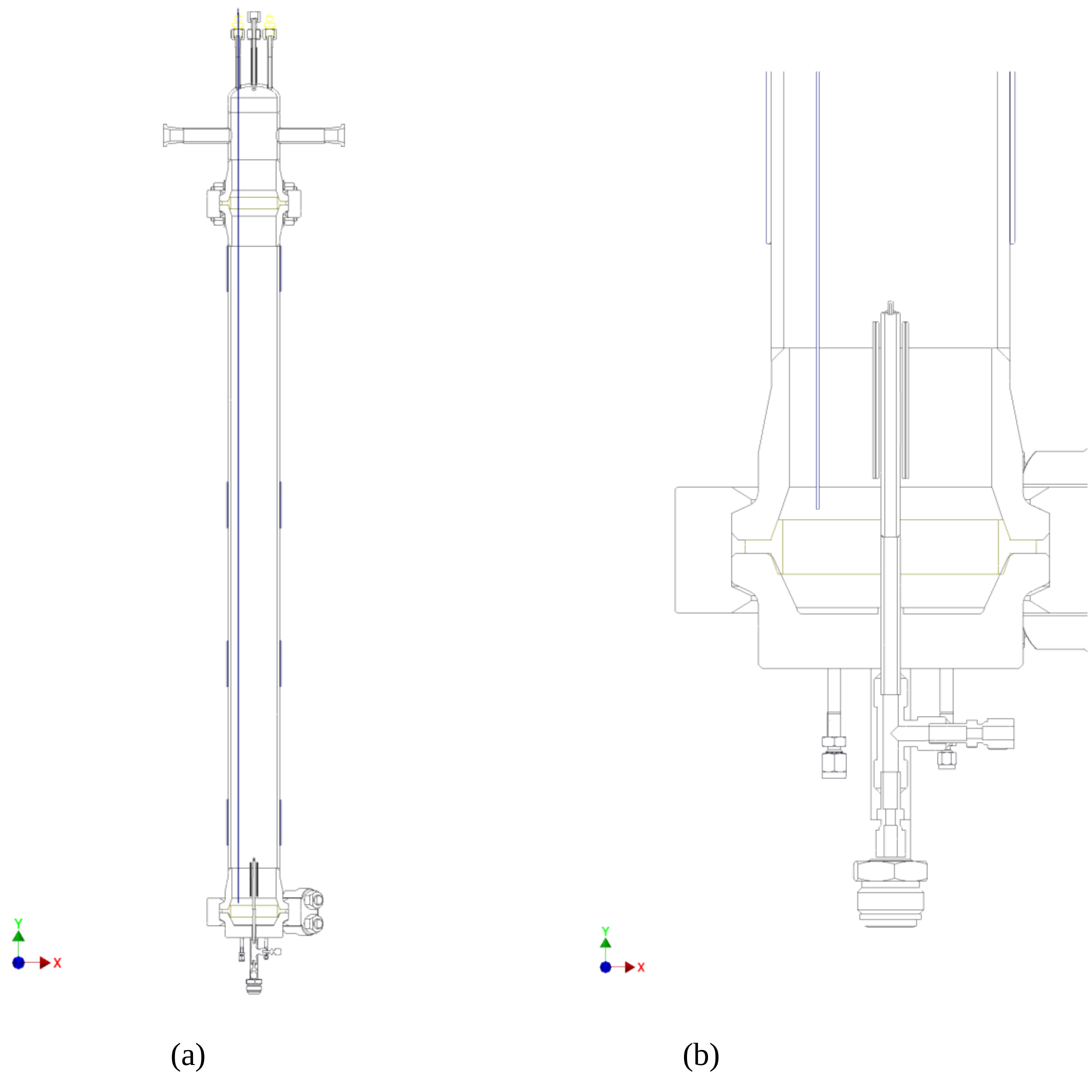


Figure 2. Drawing of Exp2018_16 test vessel and injection assembly. (a) full drawing and (b) section drawing of injector.

3.2.2 Fabricated Injector Hub

The fabricated injector hub is shown in Figure 3.

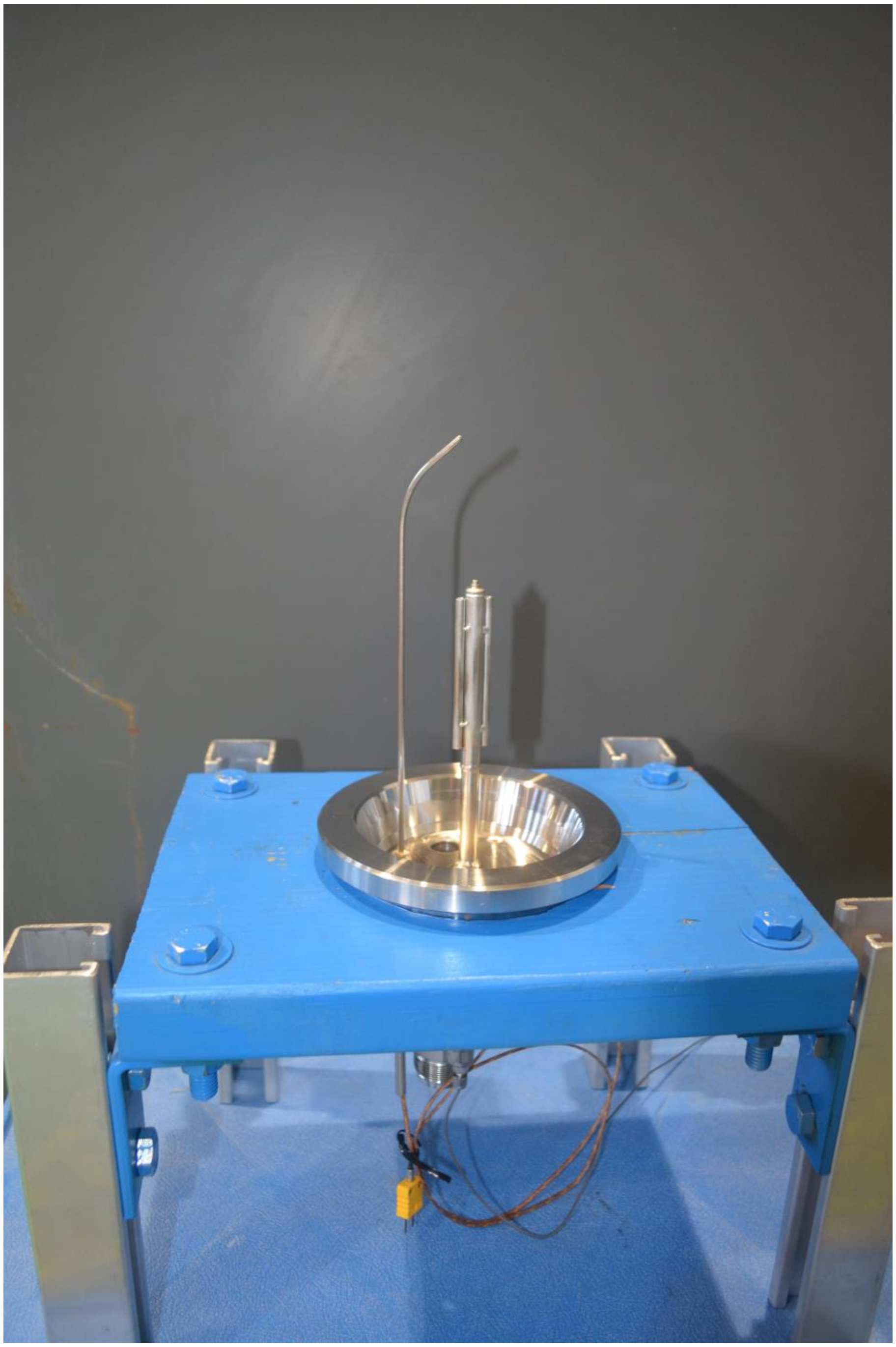


Figure 3. Picture of as-fabricated injector hub for Exp208_16.

3.3 Experiment details

The test vessel used was made by Argonne Central Shops to ASME Process Piping B31.3 specifications with 100 % radiography of welds and hydro tested to 450 psig (3.1 MPa) maximum at 1000 ºF (538 ºC) and manufactured in 2015 with a serial #CS2014_03.

A picture of the SNAKE setup for Exp2018_16 is shown in Figure 4 after insulation installation. A drawing of the as-tested test vessel and instruments is shown in Figure 5 and an updated piping and instrumentation diagram (P&ID) for Exp2018_16 in Figure 6.

Figure 4. Test vessel and SNAKE equipment for test Exp2018_16.

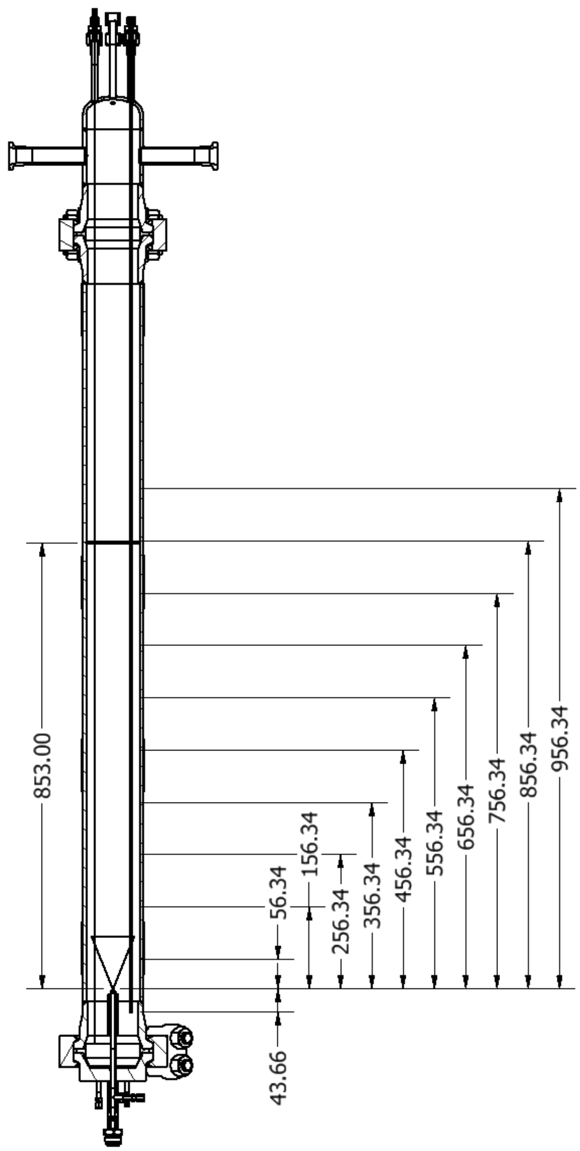


Figure 5: Test vessel, DTS, and thermocouple configuration as-tested for test Exp2018_16. Measurements are in mm. Test vessel inner diameter is 100 mm.

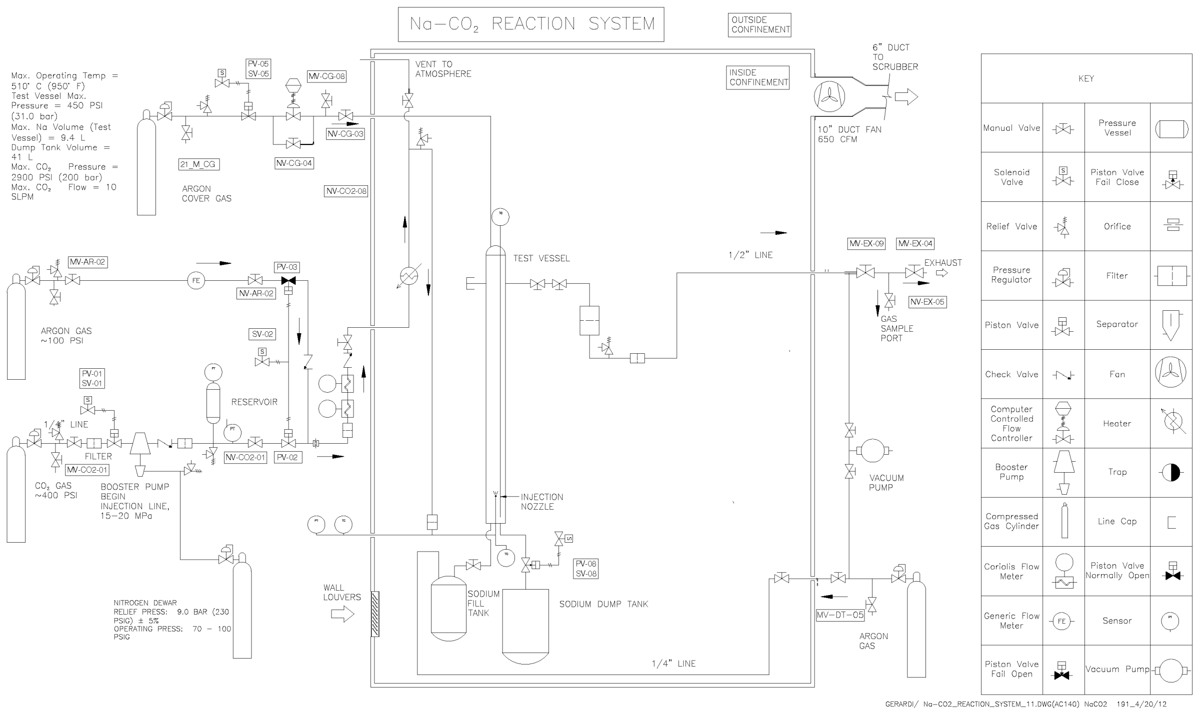


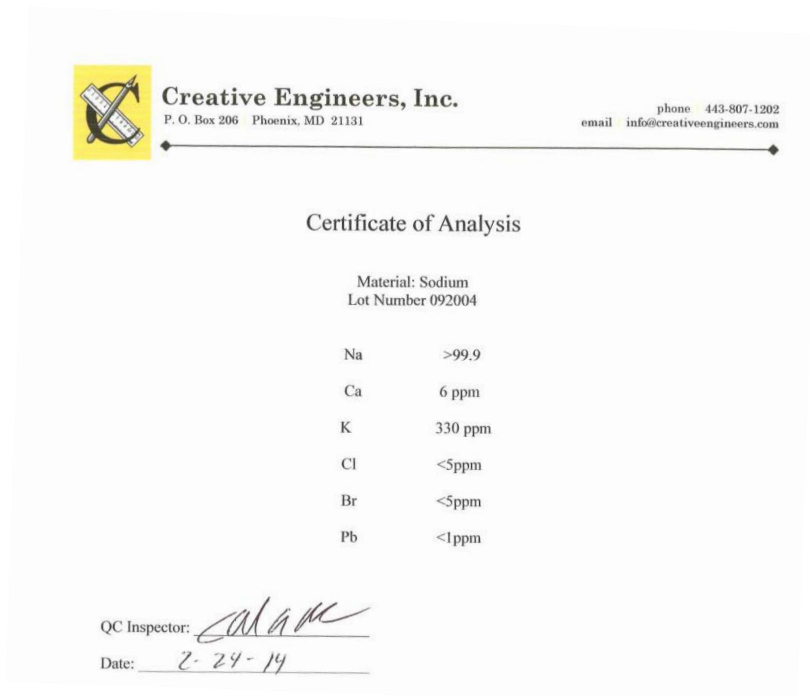
Figure 6. Process and Instrumentation Diagram for SNAKE sCO_2-sodium experiments.

The certified concentrations of the gases used in this experiment are documented in Table 2. Research Grade CO_2 was used for the nozzle injection with purities greater than 99.999%. Built-in-purifier (BIP® Air Products) argon was used for both the cover gas and pre-experiment nozzle injection.

The sodium fill tank was provided by Creative Engineers, Inc. This tank was filled with 15 kg of filtered (15 micron) R grade sodium with certifications (Figure 7).

Table 2. Certified concentrations of gases used for sodium sCO_2 interaction experiment, Exp2018_16

Gas Injected	Specified Impurity levels				
	Oxygen	**Water**	**Hydrocarbons**	**Nitrogen**	**Ar+O_2+CO**
CO_2 injected: Research Grade	n/a	< 3 ppm	< 1 ppm	< 5 ppm	< 1 ppm
Ar injected: Built-in-Purifier	< 10 ppb	< 20 ppb	< 100 ppb	< 5 ppm	n/a
Ar cover gas: Built-in-Purifier	< 10 ppb	< 20 ppb	< 100 ppb	< 5 ppm	n/a



Creative Engineers, Inc.
P. O. Box 206 Phoenix, MD 21131
phone 443-807-1202
email info@creativeengineers.com

Certificate of Analysis

Material: Sodium
Lot Number 092004

Na	>99.9
Ca	6 ppm
K	330 ppm
Cl	<5ppm
Br	<5ppm
Pb	<1ppm

QC Inspector:

Date: 2-24-14

Figure 7. Sodium certification for Exp2018_16.

Instruments used during sCO_2 injection into sodium

3.3.1 Distributed temperature sensing

The optical fiber sensing system (Gerardi et al., 2017) used for this study was an ODiSI (Optical Distributed Sensor Interrogator) Model B from Luna Innovations, Inc. (Roanoke, VA), configured to handle sensors up to 20 m in length with a 5.1 mm spatial resolution at data rates up to 50 Hz and a temperature span of -268 to 900 ˚C, though the sensor itself is likely to be restricted to a maximum temperature of approximately 600 ˚C due to the transformation from α-quartz to β-quartz. Vibration from jet noise and other experiment conditions may reduce the accuracy or data rate capabilities of this system.

The DTSs were also manufactured and assembled by Luna Innovations. Fiber optic cables were stripped of all coatings using a sulfuric acid bath to obtain bare glass fibers with 125 µm diameter. A cobalt high-temperature end termination was installed to enable sensing. Without a coating, the bare fibers are extremely fragile so they were installed into Ø 360 µm OD, Ø 160 µm OD silica capillaries and sealed.

At Argonne, the silica capillaries with internal DTS were placed into Ø 1.59 mm OD, Ø 0.056 mm ID stainless steel tubing as shown in Figure 8.

One DTS was used in Exp2018_16. It was used as a level sensor and temperature rake and extended from the vessel head downward to the bottom of the test vessel.

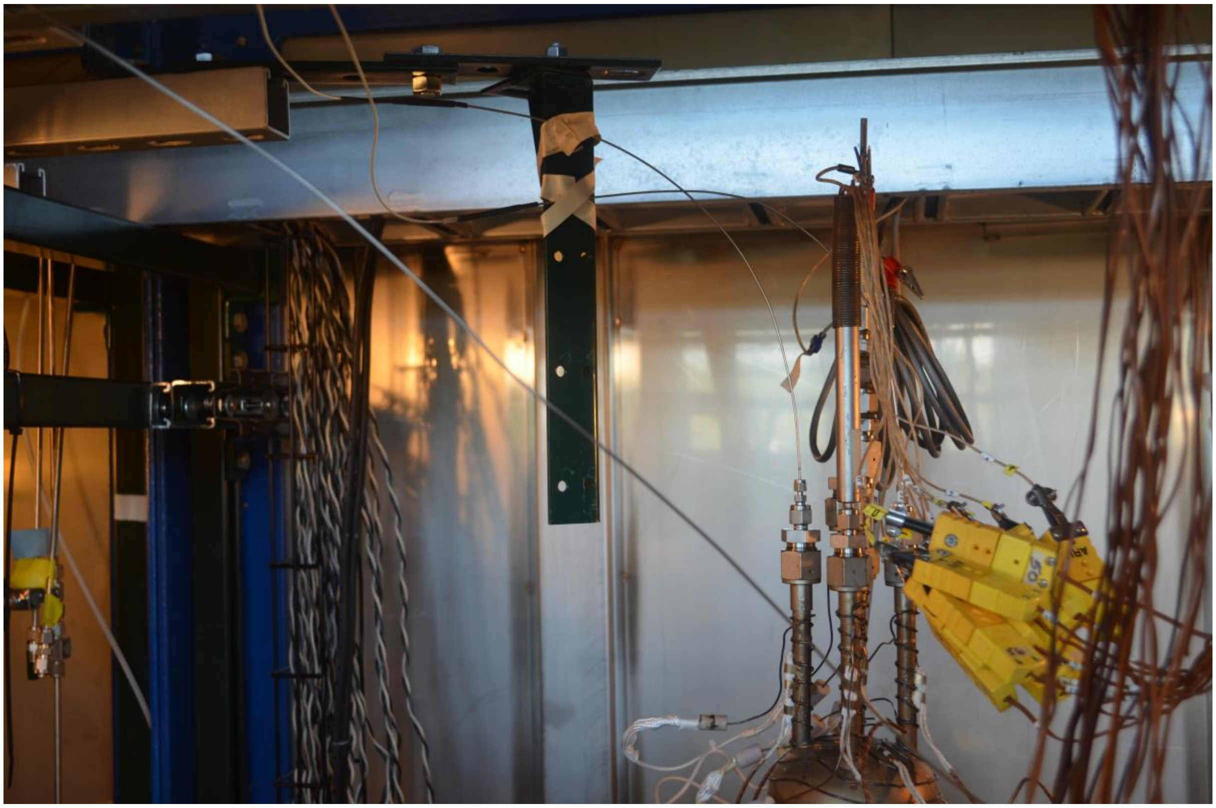


Figure 8. DTS entering stainless capillary which extends downward into SNAKE test vessel.

3.3.2 Additional instruments

A single thermocouple (Type-K) was installed in the bottom hub assembly and placed 67 mm above the injection nozzle.

Pressure measurements of the sCO_2 were made in the gas reservoir and at a location just prior to injection using a Rosemount absolute pressure transmitter with range of 0-275 bar and accuracy of ±0.1 %. The same pressure transmitter type was also used to measure pressure in the test vessel at the location of cover gas introduction into the vessel.

Supercritical CO_2 flow measurements were made by two Brooks Coriolis mass flow meters, a QMBM3 and a QMBM4. Measurement ranges and uncertainties of these flow meters are presented in Figure 9. Cover gas and exhaust flow measurements are made by Teledyne HFM-D-300 gas flow meters. A complete overview of the instruments used, their measurement ranges, and uncertainties are listed in Table 3.

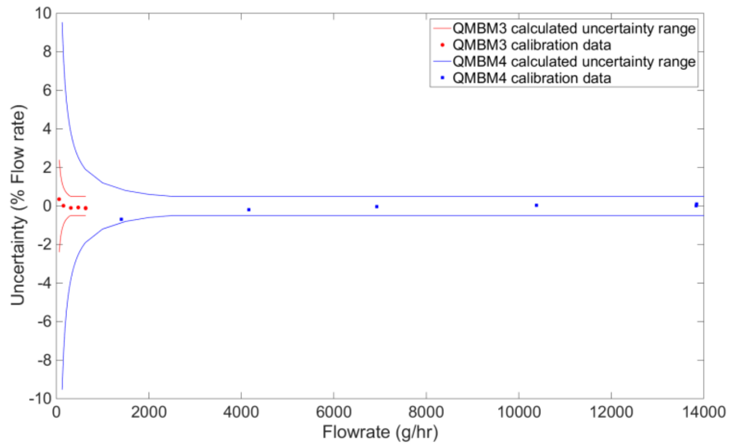


Figure 9. Comparison of measurement range and uncertainty of the two CO_2 Coriolis flow meters that are installed in series on the injection line of SNAKE.

Table 3. Major instrumentation details and uncertainty

Measured property	Location	Instrument	Measurement range	Accuracy
CO_2 injection flowrate	CO_2 injection line	Coriolis mass flow meter QMBM3	0.06-0.63 kg/hr.	±0.5 %
CO_2 injection flowrate	CO_2 injection line	Coriolis flow meter QMBM4	1-10 kg/hr.	±0.5 %
CO_2 injection pressure	CO_2 injection line	Absolute pressure transmitter	0-275 bar	±0.1 %
CO_2 reservoir pressure	CO_2 reservoir	Absolute pressure transmitter	0-275 bar	±0.1 %
CO_2 injection temperature	In-fluid at nozzle	Type-K emersion thermocouple	-200 ˚C to 1250 ˚C	±0.75 %
Ar cover gas flowrate	Ar cover gas line	Gas mass flow controller	0-25 SLM/Argon	±1 %
Ar low-pressure nozzle flowrate	Ar injection line	Gas mass flow controller	0-2 SLM/Argon	±1 %
Test vessel pressure	Top of test vessel	Absolute pressure transmitter	0-275 bar	±0.1 %
Test vessel Na level – non-intrusive	Exterior of test vessel	Gamma level meter	0-1.0 m	±0.05 m
Test vessel Na level - intrusive	Thermowell in test vessel	Distributed Temperature Sensor	0-1.5 m	±0.025 m
Test vessel Na temperature	Thermowell in test vessel	x12 Type-K emersion thermocouple	-200 ˚C to 1250 ˚C	±0.75 %
Exhaust flowrate	Gas exhaust line	Teledyne gas mass flow meter	0-30 SLM/Argon	±1 %
Exhaust gas composition	Gas exhaust line	Mass spectrometer	> 5×10^{-11} torr	±5 %

3.4 Test Procedures

3.4.1 Pre-test operations

Assembly of the SNAKE apparatus began with installation of the test vessel into the SNAKE enclosure. The test vessel consisted of a pipe section with two Grayloc butt-weld flanges. A test vessel head was installed on the top flange. The test vessel head was connected to an exhaust system, a cover gas system, and the thermocouple rake and DTS thermowell were installed. The bottom test vessel hub was then installed on the bottom of the test vessel. This bottom hub consisted of the high-pressure injection nozzle, one thermocouple measuring sodium temperature, and the bottom DTS guide tube. The bottom hub was connected to the sodium fill line, the high-pressure gas injection line, and the emergency dump line which connected to the dump tank.

Once the facility was fully assembled, pneumatic pressure testing was carried out on each subsystem to verify that the system was leak tight. This was an iterative process. Impurity removal consists of baking out the system under vacuum as high as feasible, at least 150 °C. Any fittings that may have come loose during thermal cycling were retightened. Once at room temperature, the apparatus was evacuated and back-filled with argon at least three times in order to achieve an inert argon atmosphere.

Sodium filling was carried out by heating the full system to 300 °C with an inert cover gas flow over the test vessel. A slight argon purge (10-200 psi) was maintained through the nozzle to prevent backflow of sodium. Sodium from a dedicated fill tank was introduced through the bottom of the test vessel and the level meters were monitored (gamma absorption meter, DTS level meter, and continuity meter) to achieve the desired sodium level. The DTS level meter data during the fill operation is shown in Figure 10. Filling began at approximately 0.14 hr and was complete at 0.18 hr at a sodium level of 85.3 cm above the injection nozzle. The sodium in the test vessel was then heated to approximately 400 °C and held there overnight (approximately 18 hours).

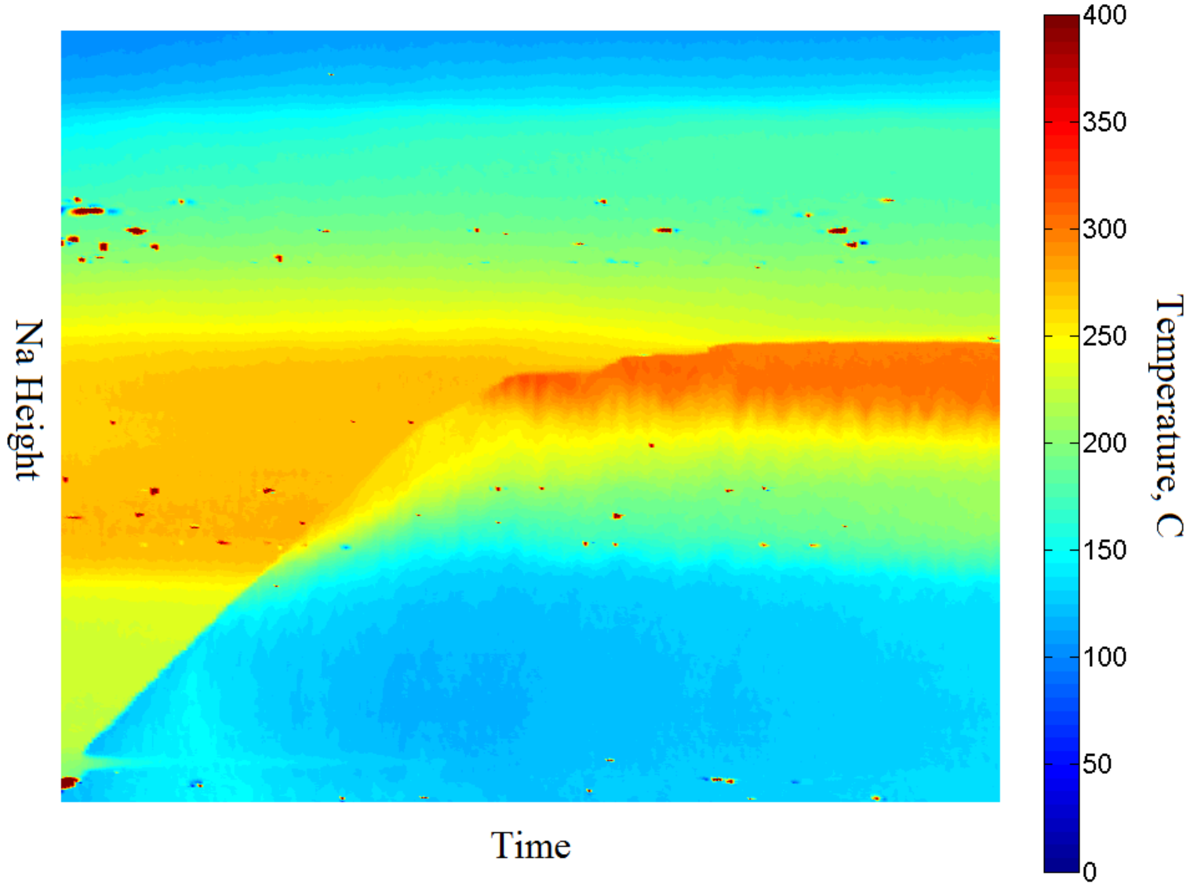


Figure 10. Distributed temperature sensor fill data showing sodium fill rate and initial sodium level.

3.4.2 Test operations

Prior to initiating the high-pressure gas injection, a fixed argon gas flow rate was established through the cover gas flow system to approximately 5-15 slpm. The high-pressure gas reservoir was pressurized to the target injection pressure.

The instruments including the acoustics, DTS, mass spectrometer, and DAS were activated prior to injection to get baseline or background data readings according to the individual instrument requirements.

The high-pressure gas line valve was then opened to initiate injection into the sodium vessel while the gas booster pump was operated in order to re-pressurize the system to the desired pressure. The line between this valve and the nozzle then quickly reached the desired injection pressure. The gas booster operation was then discontinued to allow the pressure to slowly drop as equilibrium in the injection line was reached and gas flowed into the test vessel. The instruments were monitored during the injection transient and when steady state values were reached.

3.4.3 Post-test operations

Following the experiment, the facility was cooled to room temperature with the sodium frozen in place in the test vessel.

3.5 Experiment Results of Exp2018_16

The experiment results section is broken down into several subsections. First, a description of the main thermal hydraulic measurements recorded during the test is presented and then a detailed analysis of several important measurements and observations are discussed.

3.5.1 Test overview

The initiation of sCO_2 injection began at time zero and stopped at time 0.34 hr (1230 s). Approximately 10 min (600 s) worth of data prior to sCO_2 injection is shown in most plots in order to clarify initial conditions and data is plotted until 1.0 hr after venting in order to show the thermal behavior after the experiment.

The measured flowrates of the sCO_2, argon cover gas inlet, and exhaust outlet are shown in Figure 11. Argon cover gas regulator pressure was fixed at 172 kPa and the inlet flowrate was controlled by using the cover gas mass flow controller to obtain a constant initial flow rate of 5 lpm.

The sCO_2 nozzle pressure and flowrate are shown in Figure 12. The gas booster was active for the first 3 min (240 s) in order to increase the sCO_2 pressure up to the desired starting pressure. The pressure goal of 20 MPa was not achieved due to an unexpected malfunction in the booster pressure regulator. Instead, a pressure of 15.1 MPa was achieved. The gas booster was then turned off. The sCO_2 upstream pressure slowly decreased for the remainder of the experiment as it vented into the sodium test vessel. The flowrates prior to 3 min may not reflect actual injection rates into the sodium vessel due to flow instabilities created by the boosting process. In total, approximately 58.0 g of CO_2 was injected.

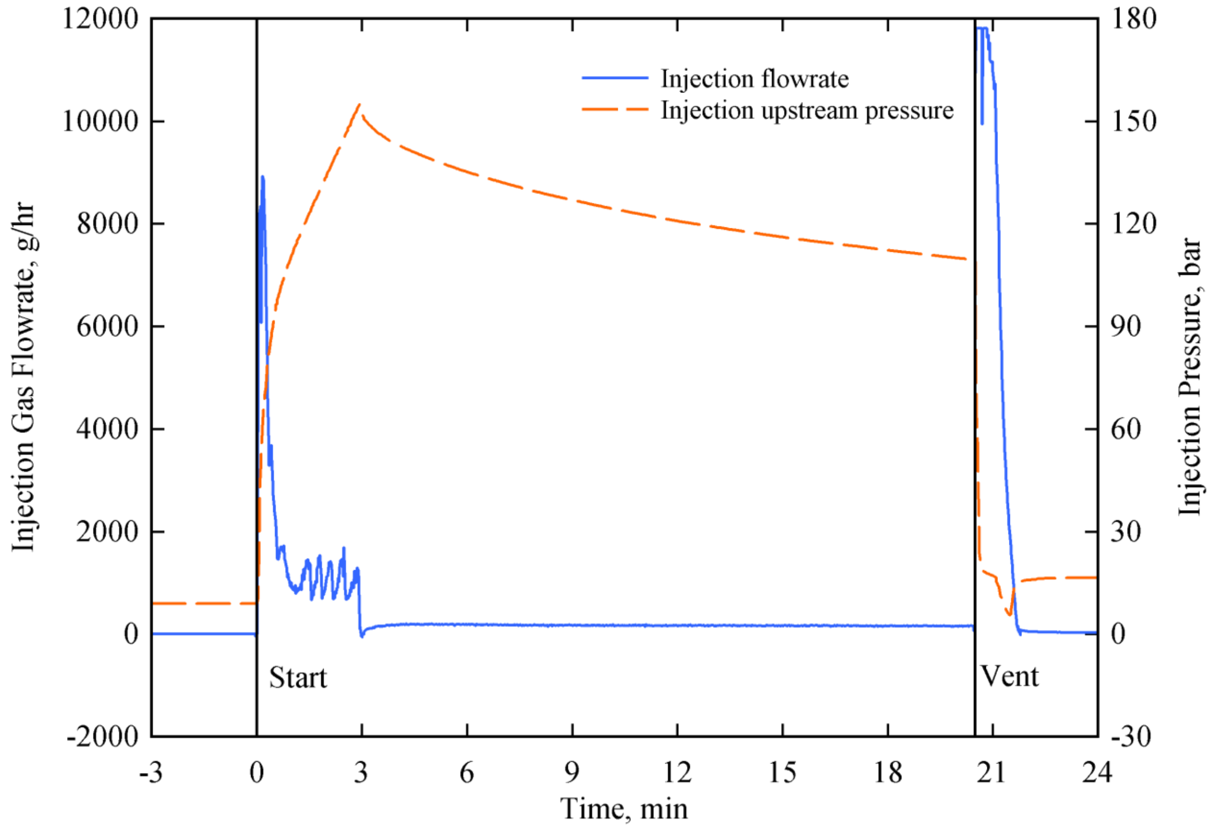


Figure 11. Flowrates and pressure of sCO_2.

The test vessel pressure, shown in Figure 12 remains between 0.115 MPa and 0.165 MPa through the test. A backpressure regulator was used to maintain a set backpressure on the test vessel. There was a small relationship between exhaust flow rate and backpressure, however. The spikes observed in the vessel pressure during the sCO_2 from injection through 20 min are small but likely real due to the sensitivity of the pressure transmitter. The large spike in pressure is due to a currently undefined phenomenon. A couple of possibilities exist, a foam block of reaction products trapped a pocket of unreacted gas or perhaps the nozzle partially blocked resulting in a delayed burst of gas when it broke free. During this entire graphed period the sodium was well above the melting temperature; therefore the only solids obstructing flow would be reaction products in some form.

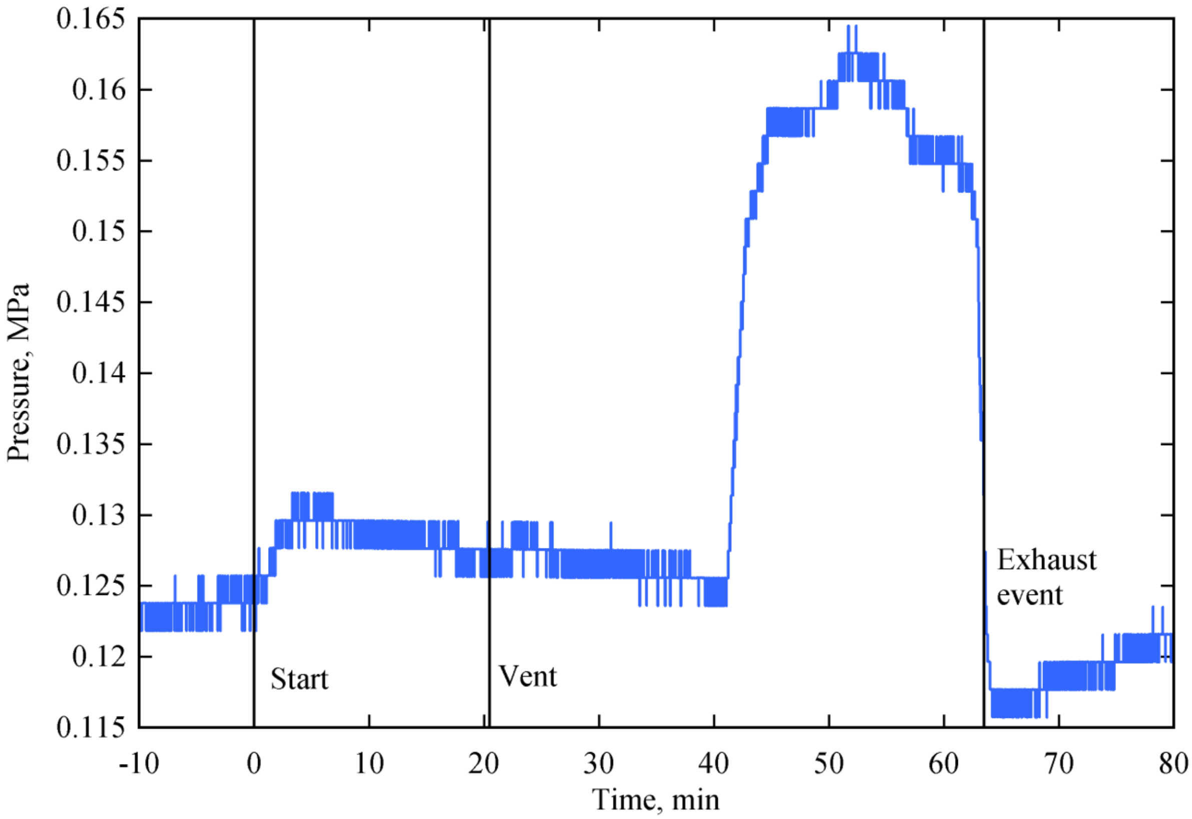


Figure 12. Test vessel pressure for sodium-sCO_2 interaction experiment.

Temperatures of the sodium column are shown in Figure 13. Temperatures along the rake are shown steady before the injection begins. No heater adjustments are performed. Once sCO_2 is injected, all thermocouples submerged in sodium show a definitive trend upwards as the exothermic reaction begins to take place. After sCO_2 venting, limited to no addition of reaction products, the temperatures begin to return to their beginning values. At the approximately 30 min mark, 10 min after injection has ceased, the heaters are started on their cooldown procedure. A further phenomenon seen earlier is shown on the thermocouple logging at the approximately 63 min mark; this corresponds with the pressure spike seen in the above figure. Another small rise in temperatures is indicative of an exothermic reaction.

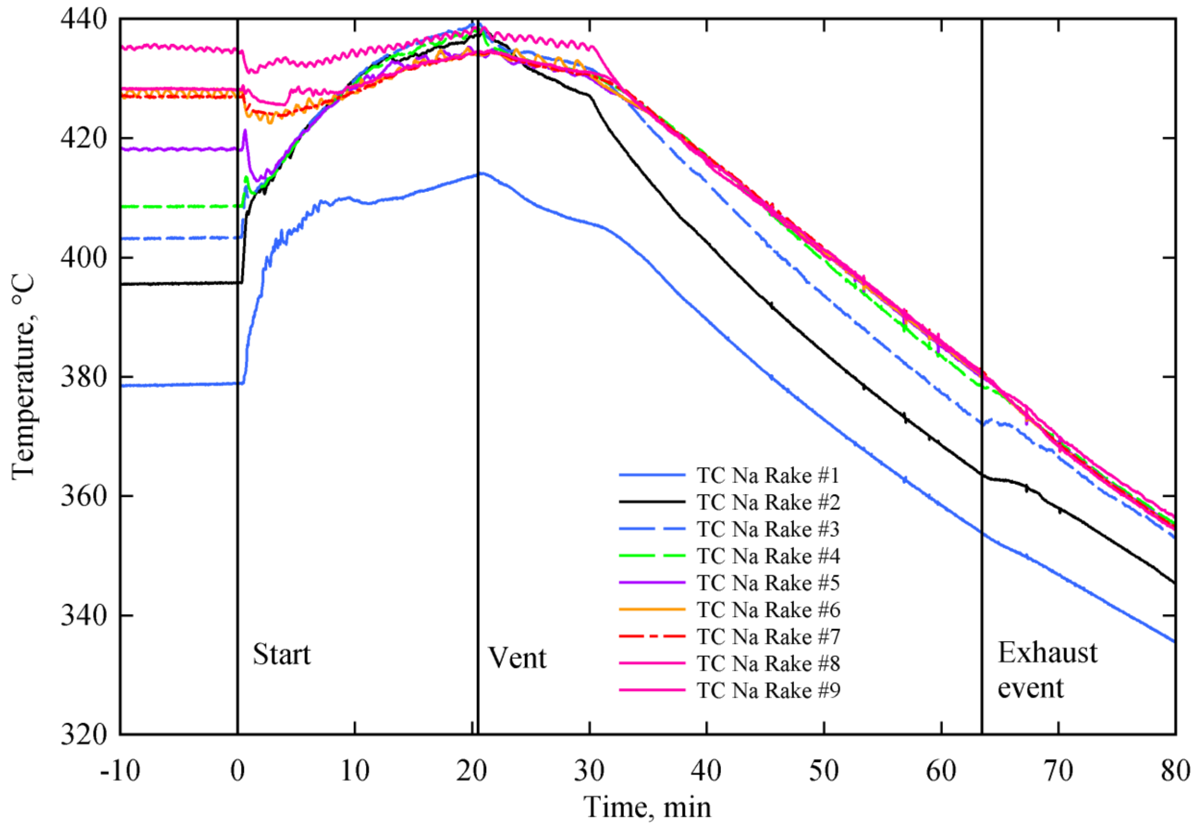


Figure 13. Temperatures recorded at the test vessel rake and sCO_2 nozzle during the sCO_2 interaction experiment.

Temperature data from the thermocouple that was placed within the sodium sCO_2 reaction cone is shown in Figure 14. Of note are the spikes in temperature appearing after venting. In the previous experiment, Exp2016_15, extreme localized temperature spikes were recorded utilizing high speed thermocouples located directly in the reaction zone. Temperatures as high as 900°C were recorded. Do to time constraints and errors in the manufacturing of the injection hub utilized in EXP2018_16, these thermocouples were omitted. However, some aspects of these extreme temperatures are shown in the readings of this TC.

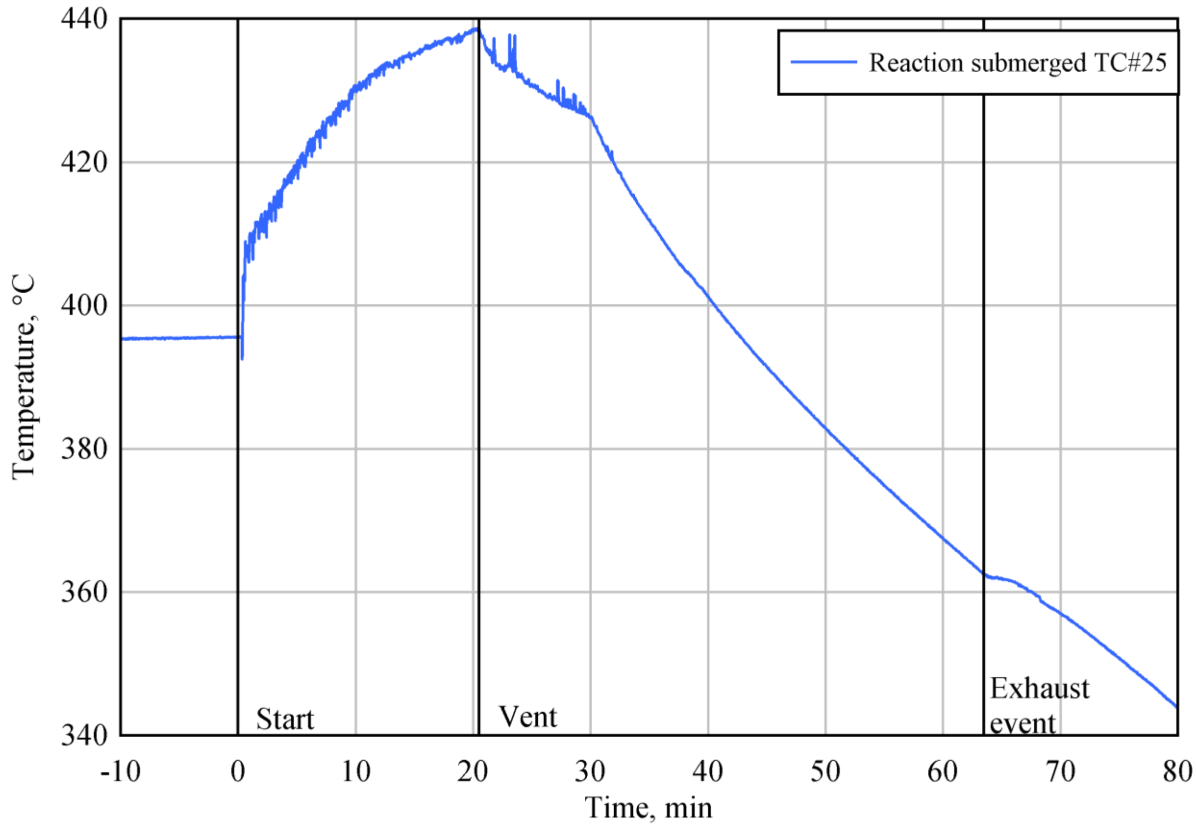


Figure 14. Thermocouple data for thermocouples placed within reaction cone.

3.5.2 Discussion and Summary

A simulated leak of sCO_2 into a sodium pool was performed using the SNAKE experiment apparatus. sCO_2 was injected into an open pool 85.3 cm deep through a Ø74 µm hole. A sustained chemical reaction between the injected CO_2 and sodium was observed. Indications of the chemical reaction included exothermic heat up, production of carbon monoxide, and consumption of CO_2.

This experiment did not result in complete blockage of the nozzle. This contrasts with two SNAKE tests performed at temperatures above 370 °C at about 500 ºC that quickly plugged the injection nozzle due to rapid production of solid reaction products. Previous studies (e.g., Gicquel et al., 2010) found that the reaction rate of sodium and sCO_2 increases dramatically with temperature. These studies also mention the possibility of a threshold temperature in the range of 450 °C – 500 °C where the reaction rate was nearly instantaneous. This could be because this experiment is below the lower threshold for this to occur. Other possibilities could be a reduced initial injection gas temperature due to the CO_2 preheater not functioning.

4 Summary

An experiment campaign was continued in Fiscal Year 2018 in the Argonne SNAKE facility that investigated the interaction between supercritical carbon dioxide (sCO_2) and sodium through injection of sCO_2 into an open surface sodium pool. Two of fifteen previous SNAKE tests revealed that self-plugging of the leakage path/nozzle occurs at higher initial sodium temperatures of about 500 ºC due to chemical reactions involving CO_2, sodium, and sodium-CO_2 reaction products. At lower sodium temperatures, the interaction phenomena involve consumption of injected CO_2 and formation of an agglomerated mass of reaction products without self-plugging. The FY 2018 test investigated interactions at an intermediate initial sodium temperature of about 411 ºC, representative of the interior of a sodium-to-CO_2 heat exchanger in a Sodium-Cooled Fast Reactor (SFR) sCO_2 Brayton Cycle energy conversion system. The experiment was successfully conducted and did not show self-plugging as evidenced by a sustained chemical reaction between the injected CO_2 and sodium and consumption of CO_2.

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